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**T. Okita, W. G. Wolfer**

Lawrence Livermore National Laboratory

**F. A. Garner**

Pacific Northwest National Laboratory

**N. Sekimura**

University of Tokyo

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**Boron Addition to Model Austenitic Steels and Void Nucleation**

T. Okita\*, W. G. Wolfer

Lawrence Livermore National Laboratory, Livermore, CA, 94550, USA

F. A. Garner

Pacific Northwest National Laboratory, Richland, WA, 99352, USA

N. Sekimura

University of Tokyo, Tokyo, Japan

\* Corresponding Author; T. Okita

7000 East Ave. Livermore, CA, 94550, USA

[TEL]+1-925-422-5909

[FAX] +1-925-423-0785

[E-mail] okita1@llnl.gov

## **Abstract**

Fe-15Cr-16Ni, -0.25Ti, -500appmB, and -0.25Ti-500appmB have been irradiated in FFTF/MOTA over a wide range of dose rate which covers more than two orders difference in magnitude, within the very limited temperature range of 387 –444 °C. The effects of dose rate and boron addition on swelling are examined.

Lower dose rates increase the swelling by shortening the incubation dose for swelling. Addition of boron does not significantly change the swelling nor the dose rate dependence of swelling for both the ternary and Ti-modified alloy.

The helium pressure of cavities is found to be much smaller than the surface tension at every irradiation condition including the lowest dose and dose rate, helium generated by boron transmutant does not play any role in cavity formation in this experiment. Cavities form without helium. The difference in cavity morphology by boron addition is most likely caused by formation of borides and by lithium.

Key word codes; H0200, N0100, S0500, and S1400

## **1. Introduction**

Natural boron contains about 20 % of the isotope  $^{10}\text{B}$ , which has an exceptionally high cross section for a (n,  $\alpha$ ) reaction. Alloying materials with boron thus provides a means to generate more helium and vary the He/dpa ratio. This, in turn, changes the incubation dose for void swelling. In addition, the neutron spectrum in the compact core of a fast reactor like FFTF changes significantly with the location of the material exposed, and so the He/dpa ratio also varies; it is higher in the peripheral regions of the core and lower in the center. Of course, this simultaneously leads to variations of the displacement rates, again higher in the center of the core and lower in peripheral regions.

As a result, it is important to take into account the concurrent effect of dose rate on the onset of swelling when studying the possible effects of helium on void swelling. To this end, we irradiated samples of simple austenitic steels side by side in FFTF/ MOTA without and with additions of 500 appm natural boron. The results for the B-doped materials are presented in this paper and compared with the results for the B-free materials. The latter have been reported earlier [1-3].

## **2. Experimental procedure**

Since a detailed description of the experimental procedures have been presented elsewhere, they are only briefly reviewed in this paper [2].

Fe-15Cr-16Ni-500appmB, Fe-15Cr-16Ni-0.25Ti-500appmB as well as the ternary alloy and Ti-modified alloy were made from the high purity elements of Fe, Cr, Ni, Ti and B by arc-melting. No precipitates were observed by TEM in the pre-irradiated samples, even though the boron concentration exceeds the solubility limit at the annealing temperature of 1050 °C.

The specimens were irradiated in the FFTF/MOTA reactor over a wide range of dose rates (from  $8.9 \times 10^{-9}$  to  $1.7 \times 10^{-6}$  dpa/sec) by placing them in different core locations. The cumulative doses ranged from 0.23 to 67.8 dpa, depending on the variation in dose rate and irradiation time. The irradiation temperatures were actively controlled and were between 387 to 444 °C; they were nearly independent of dose rate. The detailed irradiation conditions including helium generated with/without boron are shown in Table 1.

After irradiation, immersion density measurements were carried out to estimate the swelling, followed by TEM observation with 200 kV electron beam energy.

### **3. Results**

Figure 1 compares the typical cavity microstructure between B-doped alloys and non-doped alloys. In the ternary alloy and Ti-modified alloy, cavities are homogeneously distributed in the matrix, and no precipitates were observed either within the matrix or attached to cavities. In the B-doped alloys, both the size distributions and the spatial distributions are not as homogeneous, and

regions with smaller cavities are found, suggesting the presence of borides. The cavity densities are higher in B-doped alloys, while the cavity sizes are smaller.

Although cavity microstructures are affected by adding boron, the macroscopic swelling is not altered by boron addition for both the ternary alloy and Ti-modified alloy, as shown in Figure 2. More importantly, the dependence of incubation on the dose rate is preserved even in the B-doped alloys.

However, there is one exception with the B-doped ternary alloy irradiated to  $\sim 30$  dpa. A strong suppression of swelling is observed. This irradiation was conducted near the coolant inlet of the FFTF reactor core, where a stronger flux gradient exists. Within the irradiation packets, the specimens can be separated up to  $\sim 2$  cm, which results in a flux uncertainty of about a factor of two [4]. It is possible that the exceptional data point is due to this uncertainty, and not much weight should be given to this anomalous data point.

#### 4. Discussion

Helium atoms are believed to stabilize cavities by providing a gas pressure and reducing vacancy evaporation from small vacancy clusters [5]. The vacancy concentration in equilibrium with cavities is given by [6];

$$C_v^0 = C_v^{eq} \exp \left[ \frac{2\gamma}{r} - \frac{P_{He} \Omega}{kT} \right]$$

where  $C_v^{eq}$  is the thermal vacancy concentration,  $r$  is the cavity radius and  $\gamma$  is the surface energy, assumed to be  $2.0 \text{ J/m}^2$ ,  $P_{He}$  is the helium pressure of cavities,  $\Omega$  is the atomic volume,  $T$  is the temperature in Kelvin,  $k$  is the

Boltzmann's constant. The average helium pressure in cavities,  $\bar{P}_{He}$ , can be related to the total helium concentration,  $C_{He}$  in the materials, assuming negligible helium in solution. It is given by

$$\bar{P}_{He} \cdot \frac{\Delta V}{V} = 6.566 \cdot R \cdot T \cdot z \quad [MPa]$$

where  $C_{He}$  is the helium content,  $\Delta V/V$  is the swelling,  $R$  is the gas constant, and  $z$  the compressibility ratio, which is only larger than 1 for very high pressures.

Helium generation rates for the samples were evaluated by Greenwood. With the measured values for swelling, the average helium pressures in the cavities were calculated, and the results are shown in Figure 3. Figure 3 also shows the average surface tension of cavities,  $2\gamma/r$ . It is seen that for every irradiation condition in this study, the helium pressure is much lower than the surface tension of the cavities, even for the B-doped alloys. The difference is still about two orders of magnitude or more even at the lowest dose rates where the helium pressure is highest. The difference becomes even larger with increasing dose and dose rate. This clearly shows that the helium pressure is always negligible throughout the irradiation history, and hence, the cavities are voids. This also demonstrates that voids do form even in the absence of helium [7].

The difference in cavity morphology shown in Figure 1 between B-doped and non-doped samples seems to indicate a helium effect. However, boron doping is known to cause additional effects other than helium generation, such as the formation of borides and the generation of lithium. These additional effects of boron appear to modify cavity microstructure. Still, the swelling does not



change at  $\sim 400^{\circ}\text{C}$ , and the sensitivity to dose rate is preserved when boron is present.

## **5. Conclusion**

Fe-15Cr-16Ni-500appmB and Fe-15Cr-16Ni-0.25Ti-500appmB were irradiated together with the ternary and Ti-modified alloy in FFTF over a wide range of dose rate at  $387 - 444^{\circ}\text{C}$ , and the effect of dose rate and boron additions are evaluated. Adding boron does not affect swelling, and lower dose rate increases the swelling for all the alloys tested in this experiment. The helium pressure in cavities is much smaller than the surface tension of cavities even at the lowest dose rates. Therefore helium generated by boron transmutation does not play any role in cavity formation at all. However, formation of borides and lithium is found to slightly affect cavity morphology.

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**Table 1 irradiation condition in this experiment**

Dose rate, dpa/sec		Dose, dpa		Temperature, °C		appm He without B		appm He with 500appm B	
#11	#12	#11	#11& 12	#11	#12	#11	#11&12	#11	#11&12
$1.7 \times 10^{-6}$	$1.4 \times 10^{-6}$	43.8	67.8	427	408	8.96	15.5	22.3	38.2
$7.8 \times 10^{-7}$ <sup>*1</sup>	$9.5 \times 10^{-7}$	20.0 <sup>*1</sup>	32.4	390	387	6.64	10.9	19.6	31.1
$5.4 \times 10^{-7}$	$8.4 \times 10^{-7}$	14.0	28.8	430	424	3.06	5.48	14.1	22.7
$3.1 \times 10^{-7}$ <sup>*2</sup>	$3.0 \times 10^{-7}$	8.05 <sup>*2</sup>	11.1	411	410	1.84	3.22	27.5	37.6
$9.1 \times 10^{-8}$	$2.1 \times 10^{-7}$	2.36	6.36	430	431	0.45	0.70	12.2	19.0
$2.7 \times 10^{-8}$	$6.6 \times 10^{-8}$	0.71	1.87	434	437	0.23	0.35	7.84	12.6
$8.9 \times 10^{-9}$	$2.2 \times 10^{-8}$	0.23	0.61	436	444	0.013	0.021	3.59	6.10

<sup>\*1</sup>  $6.0 \times 10^{-7}$  dpa/sec and 15.6 dpa for 2cycles-irradiation specimens

<sup>\*2</sup>  $2.2 \times 10^{-7}$  dpa/sec and 5.69 dpa for 2cycles-irradiation specimens

## **Figure captions**

### Figure 1

Comparison of cavity microstructure for 2 cycles of irradiation in MOTA-2A&2B, 14.0 dpa,  $5.41 \times 10^{-7}$  dpa/sec, at 430 °C in MOTA-2A; and 14.8 dpa,  $8.4 \times 10^{-7}$  dpa/sec, at 424 °C in MOTA-2B with a cumulative dose of 28.8 dpa. Note that addition of 500 appm boron causes an increase in cavity density.

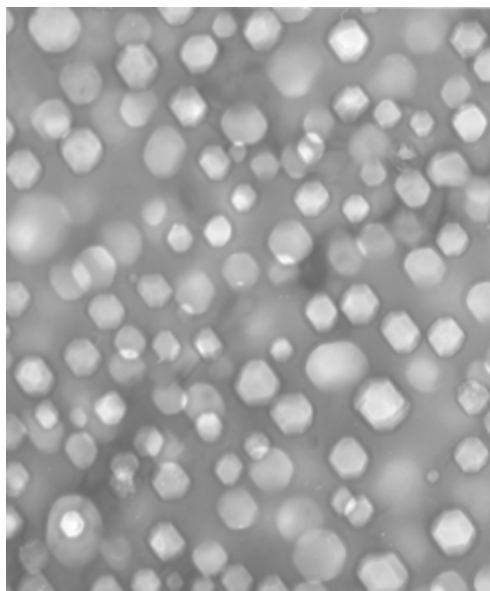
### Figure 2

Influence of 500 appm boron addition (solid symbols) on total swelling of the ternary and Ti–modified alloy. Boron addition has little effect on swelling, except for one data point derived in level1 at core edge of FFTF reactor.

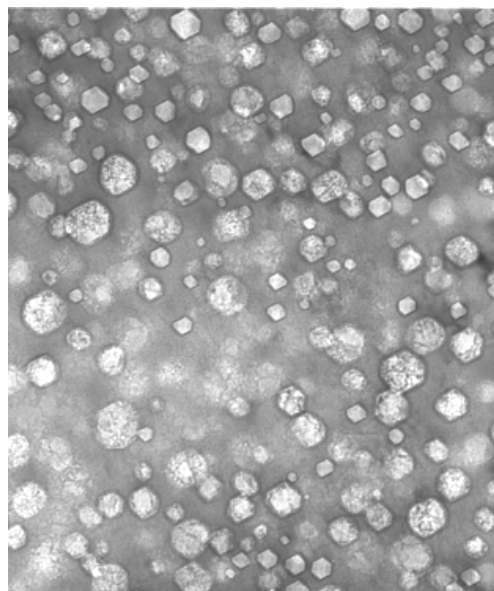
### Figure 3

Helium pressures of cavities with B-doped (solid square), without B-doped (solid circle) and surface tensions of cavities with B-doped (open square), without B-doped (open circle). Note that at every irradiation conditions, the surface tensions are much larger than helium pressures.

**Fe-15Cr-16Ni-0.25Ti**  
**5.48 appmHe**



**With 500 appm B**  
**22.7 appmHe**



**200 nm**

Figure 1 Comparison of cavity microstructure for 2 cycles of irradiation in MOTA-2A&2B, 14.0 dpa,  $5.41 \times 10^{-7}$  dpa/sec, at 430 °C in MOTA-2A; and 14.8 dpa,  $8.4 \times 10^{-7}$  dpa/sec, at 424 °C in MOTA-2B with a cumulative dose of 28.8 dpa. Note that addition of 500 appm boron causes an increase in cavity density.

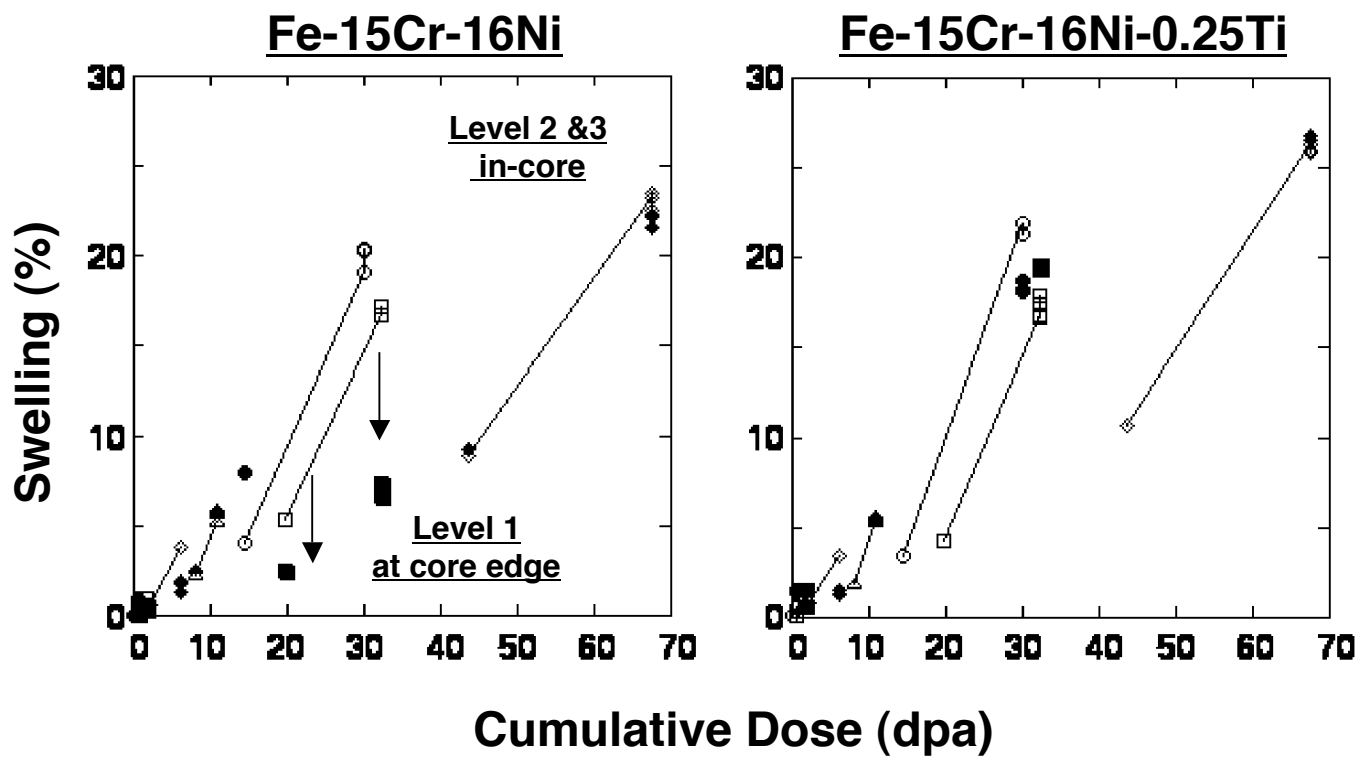


Figure 2 Influence of 500 appm boron addition (solid symbols) on total swelling of the ternary and Ti-modified alloy. Boron addition has little effect on swelling, except for one data point derived in level1 at core edge of FFTF reactor.

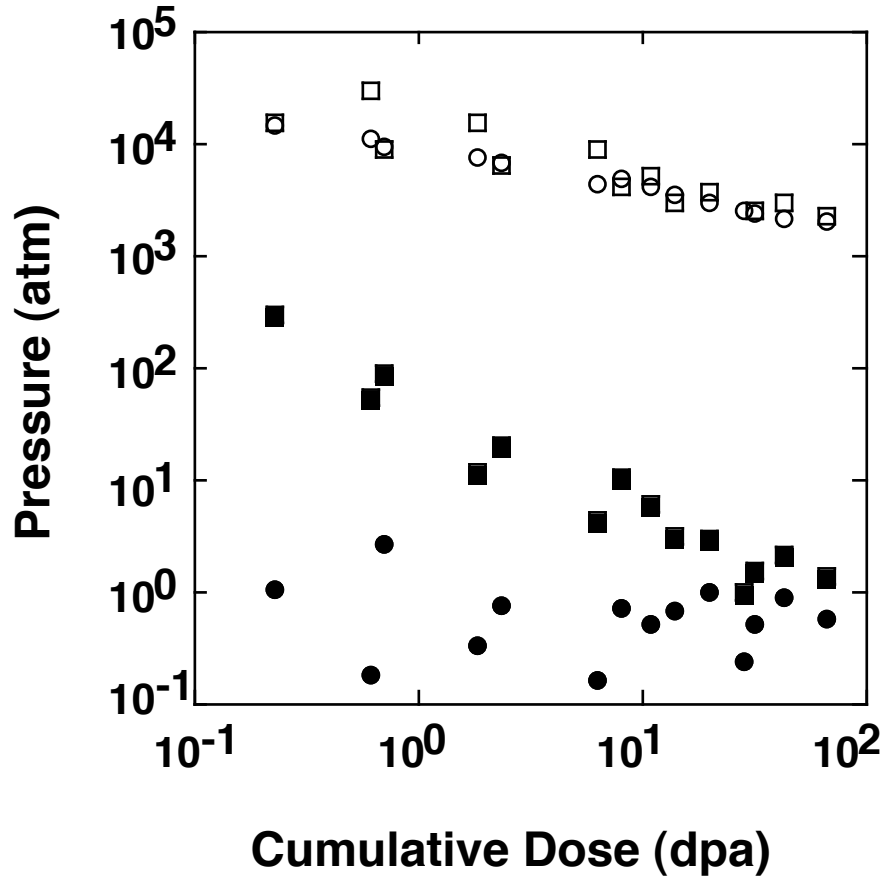


Figure 3 Helium pressures of cavities with B-doped (solid square), without B-doped (solid circle) and surface tensions of cavities with B-doped (open square), without B-doped (open circle). Note that at every irradiation conditions, the surface tensions are much larger than helium pressures.